

TITLE: THE QUANTITATION OF N-NITROSAMINES FROM TOBACCO USING
CAPILLARY GC/SINGLE ION MONITORING (SIM)/MASS
SPECTROMETRY

AUTHORS: R. F. Arrendale*, W. J. Chamberlain, and O. T. Chortyk

AFFILIATION: USDA-ARS, Tobacco Safety Research Unit, P.O. Box 5677,
Athens, GA 30613

ABSTRACT: Modern analytical methodology has been greatly aided by the rapid application of computer technology to mass spectrometry (MS). Rapid-scanning, computer-controlled quadrupole mass spectrometers are now able to accommodate high efficiency glass capillary gas chromatography (GC-2) columns. Computer-aided single ion monitoring (SIM)/MS provides detection limits two or three orders of magnitude better than traditional scanning mode MS, by spending a maximum amount of time measuring ions of known origin. Specific ions are chosen for each compound of interest. Therefore, quantitation of identified compounds can be easily achieved, even in unresolved peaks in complex mixtures. A rapid method for the analysis of N-nitrosamines in tobacco smoke by GC-2 with a nitrogen-phosphorus (NP) thermionic detector was recently developed in our laboratory. Quantitation by capillary GC/MS requires a dedicated capillary GC/MS interface. We recently modified our Hewlett-Packard 5985B GC/MS with a laboratory-constructed, open-split interface (OSI) for capillary GC/MS and will discuss its analytical benefits. Our methodology is suited to the separation of N-nitrosamines both from tobacco smoke and tobacco fractions. The GC-2 separation of the N-nitrosamines is carried out on a 25-m x 0.3-mm i.d. fused silica Superox-4 capillary column, interfaced to the MS via the OSI. The Hewlett-Packard 5985B 100-ion SIM program was used to obtain SIM/MS data. Quantitation of the N-nitrosamines was achieved by an internal standard spiking technique. Details of our methodology for the analysis of N-nitrosamines by capillary GC/SIM/MS will be discussed.

REVIEW: This work covered a lot of detail on a commonly used GC/MS linkage using fused silica capillary tubing. A minimum of instrumental alteration was required. With the computer enhancement, sensitivity is almost unlimited. 2,4-dipyridyl was used as an internal standard. Quantitation was done only on NNN because of the lack of standards for the other tobacco specific nitrosamines, namely NNK, NAB and NATB. Two ions were monitored for each amine. These were carefully selected to eliminate possible interferences. In the case of NNN, masses of 177.08 and 156 or 105 were used. A relative error of 6.7% was determined, based on twenty runs on each of five groups of samples. The author pointed out that this instrumentation was their choice over TEA for this analysis because of its capabilities in other applications.

-Reviewed by J. Millham

1000818161